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DESIGN AND CONSTRUCTION OF A LOW-FLOW, LOW-POWER TORCH FOR INDU--ETC(U)
SEP 82 R REZAAIYAAN, G M HIEFTJE, H ANDERSON N00014-76-C-0838
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A detailed study of the importance of various spatial dimensions in an ICP torch is described. Of the various dimensions which were examined, the annular spacing between the coolant (outer) and plasma (flared) tube is most critical for plasma stabilization at low argon coolant flows and applied radio frequency powers. Similarly, the inner diameter of the aerosol injection tube was found to be important, with the value of 1.0 mm chosen for a compromise between low-power, low-flow capability and routine analysis of high-solids sample solutions. A constriction at the inlet of the coolant argon was found

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20. Abstract (continued)

not to affect plasma stability greatly, but altered significantly the applied powers and coolant flows which were necessary for plasma ignition. With these and other torch dimensions optimized, it was found to be possible to ignite a plasma at a coolant flow of 5 liters per minute and an applied radio frequency power of 450 W. Continued operation of the plasma during analysis of real samples or during aspiration of a 1% NaCl solution was found to be possible alternatively at 500 W (rf power) and 3.5 L/min. coolant argon or 125 W and 5.5 L/min., respectively.

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N00014
Contract ~~N14~~-76-C-0838

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INDU/DC/GMH/TR-82-46

DESIGN AND CONSTRUCTION OF A LOW-FLOW, LOW-POWER TORCH FOR INDUCTIVELY COUPLED PLASMA SPECTROMETRY

by

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Prepared for Publication

in

APPLIED SPECTROSCOPY

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INTRODUCTION

In a relatively short time, the inductively coupled plasma has become accepted as an invaluable tool for the multielement analysis of solubilized samples.[1] However, the ICP is now recognized not to be a panacea, but has a number of limitations, many of which are currently being investigated and, hopefully, overcome. Among these limitations, the most important from a practical standpoint include the requirement that most available plasmas be operated at relatively high radio frequency powers (1-3 kW) and with rather substantial flows (12-20 L/min.) of argon coolant gas.[2]

In recognition of these limitations, a number of workers have sought to modify the ICP torch design to reduce the power and gas-flow requirements of the resulting plasma. In many of these studies, it was realized that torch modifications affect not only gas flow and power requirements, but also the ease of operation of the device, detection limits, the magnitude of the background spectrum emitted by the plasma and its stability. Early on, Genna _et al._[3] described a torch which could be ignited easily and operated at a coolant flow 30 - 40% lower than a conventional system. This reduction was achieved by incorporating a gas introduction nozzle in the coolant port; presumably, the nozzle produced a high swirl velocity which helped to stabilize the plasma and promote ignition.

Later, it was found that conventional torches could be reduced in size, modified slightly, and operated on far lower gas flows and radio frequency powers than customarily employed.[4-6] These miniaturized torches, it was reported, provided the same analytical capabilities as those of conventional size and are apparently being used now on a routine basis.[7]

Other workers[8,9] have explored the use of water-cooled plasma torches in an effort to lower the coolant argon flow. Although the resulting plasmas appeared stable, even at total argon flows of 2 L/min., analytical performance (detection limits, interferences) appeared to be substantially poorer than in competitive systems. Also, both the water-cooled and miniaturized torches require modifications to conventional ICP power systems, ordinarily in the design of the load coil used to couple RF energy into the plasma.

Clearly, what would be desired is a plasma torch of the size usually utilized (20 mm o.d.), but with the arrangement of concentric quartz tubes designed to permit operation at reduced rf power and coolant gas flow levels. The feasibility of such high-efficiency torches was first demonstrated at the Sherritt-Gordon Laboratories. Using those torches as a model, a systematic study has now been undertaken to determine the dimensions of the plasma torch which are most critical; this study is reported herein.

In this investigation, a novel approach was employed to assess torch performance. In this approach, "plasma stability" curves were developed which delineate the minimum rf power-coolant gas flow levels which are capable of sustaining an ignited discharge. To obtain each curve, an ignited plasma is first allowed to stabilize, after which the coolant argon flow or radio frequency power is decrementally lowered until the plasma spontaneously quenches. The collection of such quenching points, when plotted on a power-flow axis system, form a continuous curved line which delineates stable plasma operation. The effect of each torch dimension on the stability curve can then be determined readily.

The result of this investigation has been an "optimized" ICP torch which is capable of supporting a stable discharge at coolant gas flows as low as

3.5 L/min. and at radio frequency powers down to 125 W. Ignition of the plasma with the new torch is possible at a power of 450 W, but requires a coolant flow of approximately 11 L/min. Importantly, these same conditions can be employed even when high-salt content (1% NaCl) solutions are sprayed into the discharge. Evidence of the utility of the system for the analysis of real samples is provided through examination of an NBS standard reference material (#1573, tomato leaves).

I. EXPERIMENTAL

Power to the modified torches was supplied by a 2.5 kW, 27.12 MHz crystal-controlled generator (model HFP-2500D, Plasma-Therm, Inc., Kresson, NJ) with an automatic impedance matching unit (model ANM-2500E, Plasma-Therm, Inc., Kresson, NJ). A three-turn water-cooled load coil of conventional dimensions was constructed from 1/8 inch (o.d.) copper tubing. The gas handling system was identical to the one previously described,[4] except that a concentric (Meinhard) nebulizer was utilized. In all experiments, the nebulizer and plasma (intermediate) argon flow rates were held at 0.5 L/min. Unless otherwise noted, all measurements were made with water aspirated into the plasma at a flow rate of 1 mL/min. The optical and readout systems were also as previously described.[4]

The design parameters and dimensions of the torch which were optimized are indicated in the expanded torch diagram of Fig. 1. Specifically, these parameters were: A. the annular spacing between the outer (coolant) tube and the flared-out portion of the intermediate (plasma) tube; B. the diameter of the orifice used for injection of coolant gas into the torch; C. the length of the flared-out portion of the plasma tube; D. the length of the coolant tube which extends beyond the plasma tube; E. the distance by which the aerosol

injection tube is recessed below the plasma tube; and F. the inner diameter of the orifice used for injection of aerosol into the plasma. For clarity, a complete schematic view of a typical plasma torch is also shown in Fig. 1; none of the dimensions in Fig. 1 is meant to illustrate those ultimately adopted in the optimized device.

To ascertain the optimal values of each of the design parameters indicated in Fig. 1, plasma stability curves were obtained for a number of torches, in which only the parameter being investigated was changed. The optimal value of that parameter was then incorporated into the torches used to investigate subsequent parameters; parameters were investigated in the alphabetical order shown in Fig. 1.

Because of the possible interaction of the effects of some design variables, the entire design sequence was repeated. Importantly, very little change in the final dimensions was produced by this second iteration. After adoption of the final, optimized set of dimensions, several torches were constructed to those specifications and their stability curves compared. The curves were reproducible within expected experimental variability.

Stability curves were obtained in the manner described in the introduction to this paper. However, it is important to recognize the necessity of careful monitoring of the plasma torch when coolant gas flow is decremented. Under the aerosol and plasma conditions employed in this study, no problems with torch melting were observed. However, earlier studies at the Sherritt-Gordon Research Laboratory suggested that torch melting occasionally occurred and a judicious hand at the radio frequency power controls would be prudent if these studies are to be repeated.

The 1% NaCl solution was prepared from the reagent grade salt. The sample of NBS standard reference material 1573 (tomato leaves) was prepared in the

customary way;[10] five replicate determinations of selected elements in the SRM were performed using conditions found favorable for the optimized torch (450 W of radio frequency power and 5 L/min. of argon coolant).

II. RESULTS AND DISCUSSION

Figure 2 shows the effect of the annular spacing between the plasma and coolant tubes (cf. A on Fig. 1). Obviously, this dimension has a pronounced effect on the power and flows required to sustain a plasma; 0.5 mm appears to be the optimal spacing. Presumably, a smaller annular spacing increases both the vertical and tangential component of the coolant gas flow and directs it more strongly against the coolant tube. As a consequence, plasma performance and sustenance will be possible at lower flows.

The effect of annular spacing on the required radio frequency power is less obvious. It has been reported[11] that higher swirl velocity creates a low-pressure region above the plasma tube, which tends to stabilize the discharge; moreover, high velocity also produces tighter ionization spirals, which enhance ionization. Both factors would enhance plasma sustenance at low RF powers.

Constricting the inlet tube employed for coolant gas should also increase swirl velocity for any given flow of coolant. However, as shown in Fig. 3, very little improvement in plasma stability is produced by constricting the inlet tube from a relatively large value (4 mm) considerably (to 1 mm). However, the constricted inlet port does influence substantially the RF power and coolant flow required to ignite the plasma, as shown in Fig. 4. The curves shown in Fig. 4 were obtained in a manner similar to that employed for Figs. 2 and 3; however, plasma ignition rather than sustenance was the determining criterion in defining the group of data points.

Clearly, a constricted inlet tube critically affects plasma ignition but has little influence on the stability of an existing discharge. With no constriction, powers approaching 1 kW and coolant flows of approximately 15 L/min. are needed for plasma ignition; in contrast, a plasma torch with a 1 mm coolant inlet tube permits ignition at powers of 500 W or less and coolant flows near 10 L/min.

One might surmise from Figs. 3 and 4 that plasma sustenance and ignition might be related, respectively, to the vertical and horizontal velocity components of the coolant gas flow. Recent reports[12] indicate that the ratio of vertical-to-horizontal velocities in the coolant gas is approximately 10:1, indicating that the former velocity would be most strongly affected by annular spacing (cf. Fig. 3). Understandably, this vertical velocity component would be most directly responsible for creating a low-pressure region at the torch outlet and would therefore stabilize the plasma. In contrast, a constricted inlet tube would most greatly effect the horizontal component of the coolant gas velocity; the resulting tighter ionization spiral, which supposedly enhances ionization[11] would thus simplify plasma ignition. The constricted tube was adopted for subsequent torch designs.

The optimal length of the flared portion of the plasma (intermediate) tube is approximately 15 mm as indicated in the plasma stability curves of Fig. 5. One would assume that an excessively long flared section would exert a drag on the flowing gases and, at a given supply pressure, produce a slightly slower exit velocity for the coolant gases. However, a greatly shortened (10 mm) flared section might not allow a smooth gas flow pattern to develop, thereby producing slight eddies or turbulence in the exit flow pattern.

An extended coolant tube has very little influence on discharge stability, as shown in Fig. 6. This unsurprising finding is nonetheless significant because of the desirability of employing extended coolant tubes to reduce spectral background[13,14] or to enable optical coupling of the ICP with a vacuum monochromator.[15]

A number of individuals[3,4,6] have found that a capillary tip attached to the aerosol injection tube (cf. Fig. 1) enhances aerosol penetration of the plasma. Figure 7 illustrates the effect of such a capillary tip and its recession below the flared plasma tube on plasma stability. From Fig. 7, plasma stability appears to be greatest with the aerosol tube recessed 5 mm below the plasma tube; a smaller or greater recession (3 or 10 mm) destabilizes the discharge somewhat. However, an extended-length capillary and short capillary behaves similarly as far as plasma sustenance is concerned. These kinds of aerosol tubes correspond, respectively, to those illustrated in diagrams (b) and (d) in Fig. 2 of reference 16.

The inner diameter of the aerosol injection tube has an even greater effect than its distance from the plasma fireball as revealed by the plasma stability curves of Fig. 8. A relatively large diameter (2 mm) on the aerosol injection tube apparently perturbs the plasma much more greatly than the stream issuing from a narrower orifice (0.75 or 1 mm). However, below an injection orifice of 1 mm, very little further improvement is noted. For this reason, and to reduce encrustation of the aerosol injection tube during nebulization of organic-containing or high-salt content solutions, a 1 mm injection tube diameter would be preferred for most applications and was adopted for use in the final "optimized" torch.

The torch's performance as illustrated in Fig. 8 was considered "optimized" for the purposes of the present study. For this optimized torch, values for the various dimensions illustrated in Fig. 1 are cited in Table I. Using these dimensions as a starting point, the entire optimization procedure outlined above was repeated, with no appreciable changes in the optimal parameters being obtained. Accordingly, this "optimized" configuration was then tested for use in practical applications.

In many such practical situations, it is necessary to determine elemental concentrations in samples of high salt content. To illustrate the ability of the new plasma torch to handle such samples, plasma stability curves were obtained for a discharge into which was aspirated a 1% NaCl solution. This curve is reproduced in Fig. 9 and is compared to the stability curve developed during aspiration of a distilled water solution. The congruency of the curves verifies the utility of the new torch for the examination of real samples.

The final validation of the utility of the new torch is found during analysis of a real sample, in the present case an NBS standard reference material (#1573, tomato leaves). For this analysis, the plasma was ignited at a forward power of 450 W and a coolant flow of 11 L/min. However, the tomato-leaves analysis was performed at the same forward power, but with a coolant flow of only 5 L/min. As during the construction of plasma stability curves, plasma and aerosol injection flows were both 0.5 L/min.; the same concentric nebulizer was employed as described earlier and the sample uptake of 1 mL/min was supplied by means of a peristaltic pump. No "releasing agents" or other additives were included with the sample. The results of this analysis, shown in Table II for five trials of the sample, compare very favorably with the values certified by NBS.

III. CONCLUSIONS

From Figs. 8 and 9 and Table II, the new plasma torch described in this study is able to perform analyses of real samples at unusually low coolant flow rates and applied radio frequency powers. Although the power and flow values employed in the real-sample analysis were already dramatic, it should be noted from Figs. 8 and 9 that alternative analysis conditions could be utilized.

In particular, most workers who currently employ plasma emission spectroscopy have available relatively high-power radio frequency supplies, so that savings in argon flow would be of greatest benefit. The conditions chosen in this study for the analysis of tomato leaves indicate that flows as low as 3.5 L/min. could be employed in such cases. However, Figs. 8 and 9 reveal that even lower rf powers could be employed if higher coolant flows were tolerable. Specifically, an alternative set of analysis conditions would require only 125 W of rf power but at least 5 L/min. of coolant argon.

The immediate importance of these findings is that it should be possible for current users of ICP instrumentation to realize substantial savings in coolant argon consumption simply by adopting the new torch design. However, for the longer term, it should also be possible to develop new power systems for ICP application which operate on solid-state drivers. Such modified supplies would not only be more compact and energy-efficient, but they could be supplied from standard 110 volt power outlets and could possibly employ active impedance-matching systems. The new units would therefore be readily portable and more easily adopted to "bench-top" use.

ACKNOWLEDGEMENT

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Table I. Optimized ICP Torch Dimensions for Low-Power, Low-Flow Operation

Parameter	Designation in Fig. 1	Optimal Value (mm)
Annular Spacing Between Plasma and Coolant Tubes	A	0.5
Coolant Inlet Port Diameter	B	1
Length of Flared Portion of Plasma Tube	C	15
Length of Coolant Tube Extending Beyond Plasma Tube	D	22
Recession of Aerosol Injection Tube Below Rim of Plasma Tube	E	5
Inner Diameter of Aerosol Injection Capillary Tube	F	1

Table II. Results of ICP Analysis of NBS SRM 1573 (Tomato Leaves) Using New Low-Flow, Low-Power Torch[a]

Element	Wavelength (nm)	Concentration, this study (mg/g)[b]	NBS Certified Value (mg/g)
Ca	393.4	29.5 ± 0.5	30 ± 0.3
Fe	258.2	0.68 ± 0.04	0.69 ± 0.025
Mn	257.6	0.246 ± 0.012	0.238 ± 0.007
Cu	324.7	0.016 ± 0.005	0.011 ± 0.001
K	404.7	41.5 ± 5.1	44.6 ± 0.3
Sr	407.7	0.0445 ± 0.0002	0.0449 ± 0.0003
Zn	213.9	0.057 ± 0.018	0.062 ± 0.006

[a]Analysis conditions: Coolant flow = 5 L/min; applied rf power = 450 W; plasma and aerosol Ar flows = 0.5 L/min

[b]Results of 5 determinations

FIGURE LEGENDS

Figure 1. Cutaway views showing typical ICP quartz torch (right) and expanded view of the same torch (left). Dimensions of torch labeled alphabetically are those optimized in the present study for low-power, low-flow operation.

Figure 2. Plasma stability curves illustrating the effect of the annular spacing between the flared portion of plasma (intermediate) and coolant (outer) quartz tubes. ✱: 1 mm annular spacing; *: 0.7 mm annular spacing; □: 0.5 mm annular spacing. Length of coolant tube: 20 mm; length of flared portion of plasma tube: 20 mm; spacing between aerosol and plasma tubes: 2 mm; aerosol tube inner diameter: 1.5 mm.

Figure 3. Plasma stability curves illustrating the effect of a constriction in the coolant inlet tube. *: straight inlet tube with i.d. of 4 mm; ○: constriction of coolant inlet tube of 1 mm i.d.; length of coolant tube: 22 mm; length of flared portion of plasma tube: 20 mm; distance between aerosol and plasma tubes: 5 mm; inner diameter of aerosol tube: 1.5 mm; annular spacing between plasma and coolant tubes: 0.7 mm.

Figure 4. Effect of coolant inlet tube constriction on ease of ignition of ICP discharge. *: straight coolant tube with i.d. of 4 mm; ○: inlet tube constricted to 1 mm i.d. Annular spacing

between coolant and plasma tubes: 0.5 mm; length of coolant tube: 22 mm; length of flared portion of plasma tube: 15 mm; distance between aerosol and plasma tubes: 5 mm; inside diameter of aerosol tube: 1 mm.

Figure 5 Plasma stability curves illustrating the influence of the length of the flared portion of plasma tube. ○ : flared length = 10 mm; * : length = 15 mm; □: length = 25 mm. Annular spacing between plasma and coolant tubes: 0.5 mm; length of coolant tube: 22 mm; distance between aerosol and plasma tubes: 5 mm; inside diameter of aerosol injection tube: 1 mm.

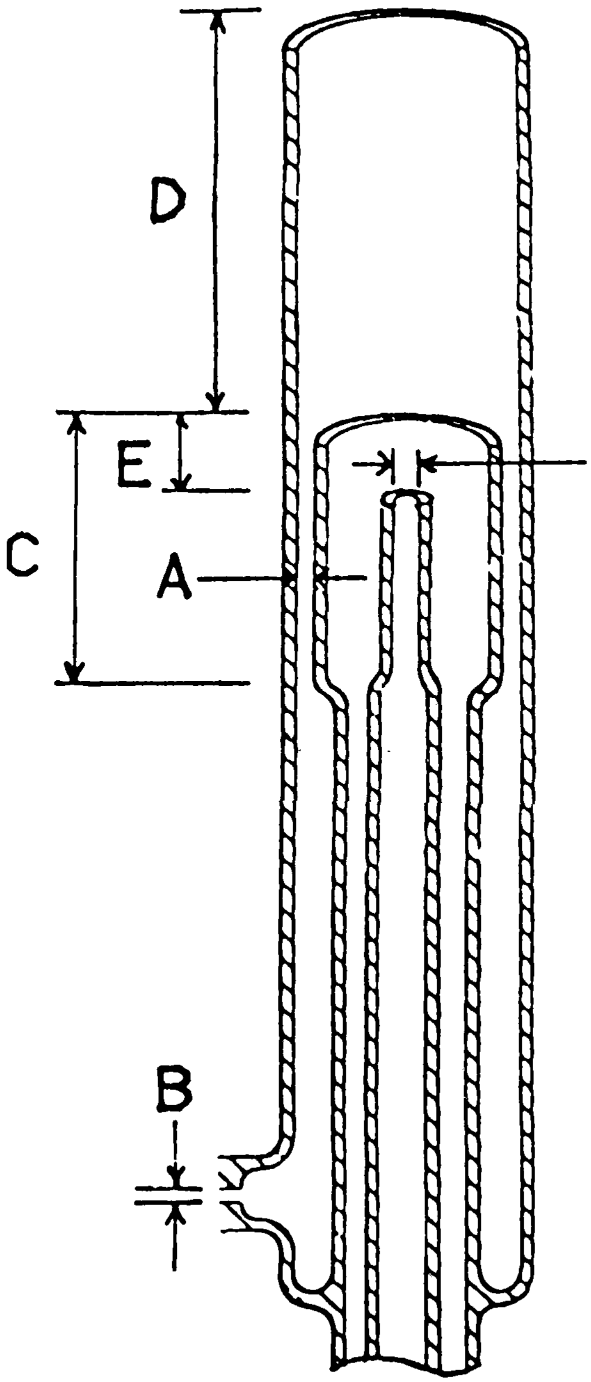
Figure 6 Effect on plasma stability curves of coolant tube extension beyond rim of plasma tube. △ : coolant tube extension = 20 mm; □: coolant tube extension = 22 mm; ○: coolant tube extension = 30.5 mm. Annular spacing between coolant and plasma tube: 0.7 mm; length of flared portion of plasma tube: 15 mm; distance between aerosol and plasma tubes: 5 mm; inner diameter of aerosol injection tube: 1.5 mm.

Figure 7 Plasma stability curves illustrating the effect of length of capillary injection tube and its recession below rim of plasma tube. □: long ("straight") aerosol tube terminated 3 mm below plasma tube rim; * : straight aerosol tube terminated 5 mm below plasma tube rim; △: short (tapered) aerosol tube

terminated 5 mm below plasma tube rim; ○: straight aerosol tube positioned 10 mm below plasma tube rim. Annular spacing between plasma and coolant tubes: 0.5 mm; length of flared portion of plasma tube: 15 mm; coolant tube extension beyond plasma tube: 22 mm; inside diameter of aerosol injection tube: 1 mm.

Figure 8. Effect of inside diameter of aerosol injection tube on plasma stability curves. ✱: inner diameter = 0.75 mm; *: inner diameter = 1 mm; △: inner diameter = 2 mm. Annular spacing between plasma and coolant tubes: 0.5 mm; length of flared portion of plasma tube: 15 mm; extension of coolant tube beyond plasma tube: 20 mm; distance between aerosol and plasma tubes: 5 mm.

Figure 9 Comparison of plasma stability curves for discharge into which is aspirated a distilled water solution (*) and a 1% NaCl solution (○).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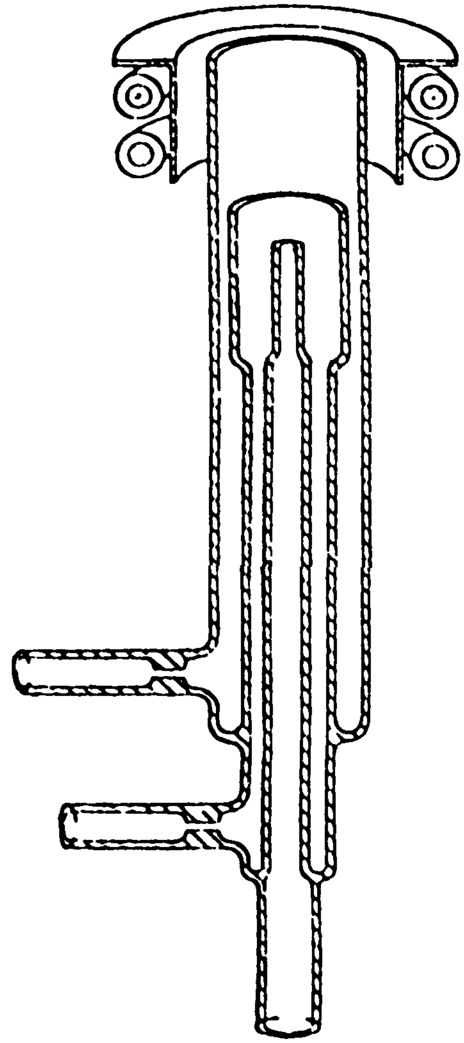

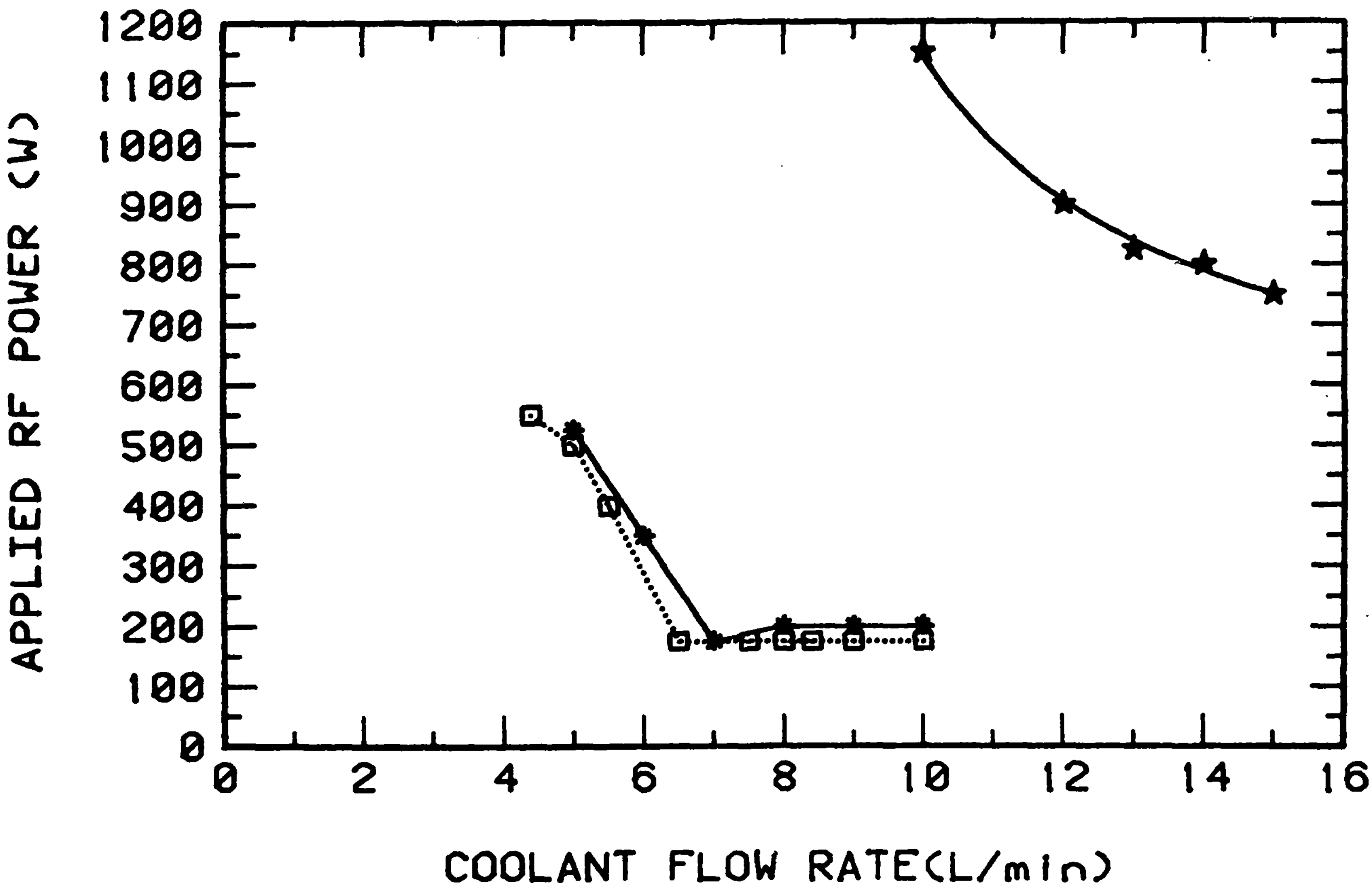
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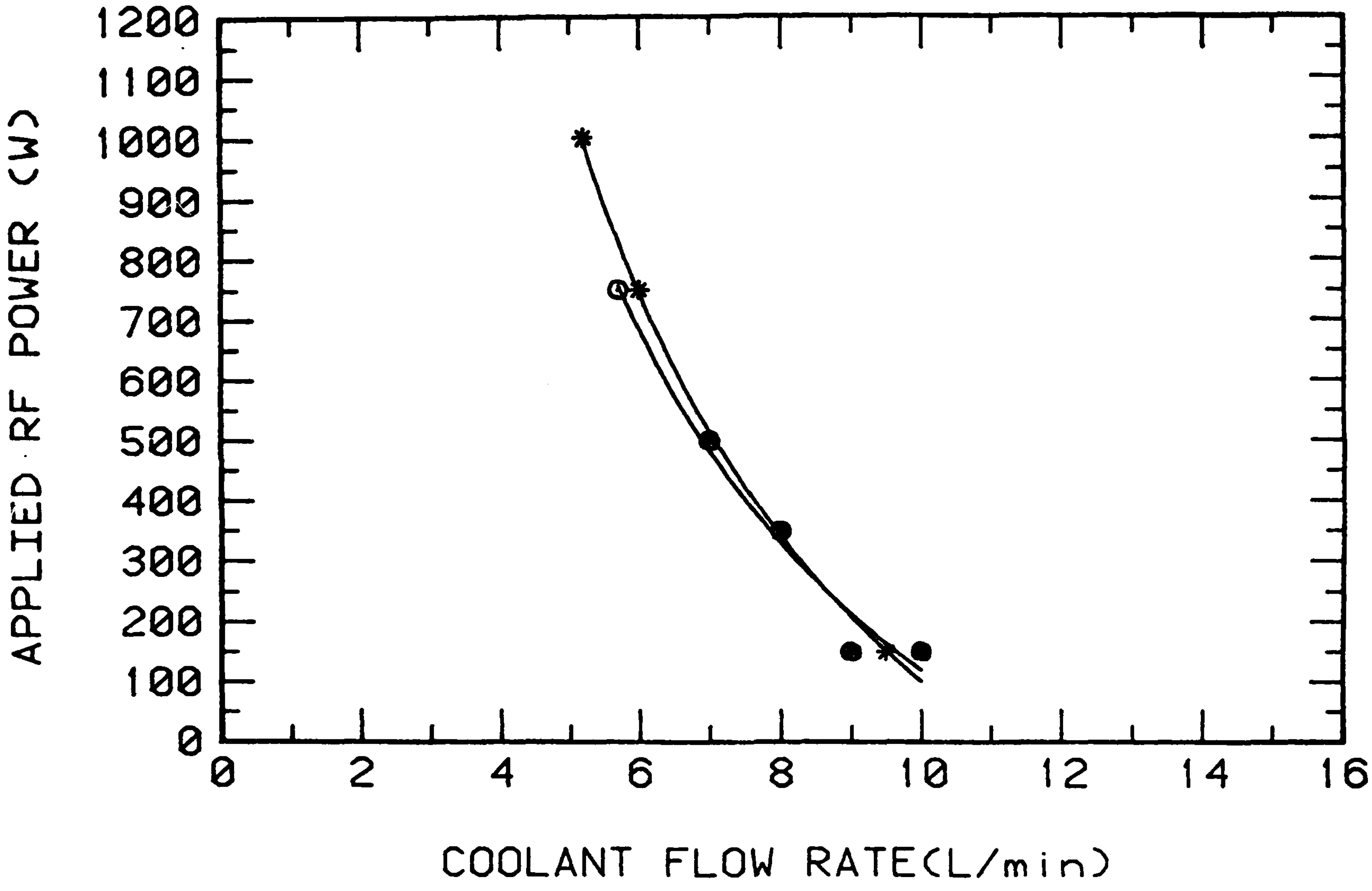
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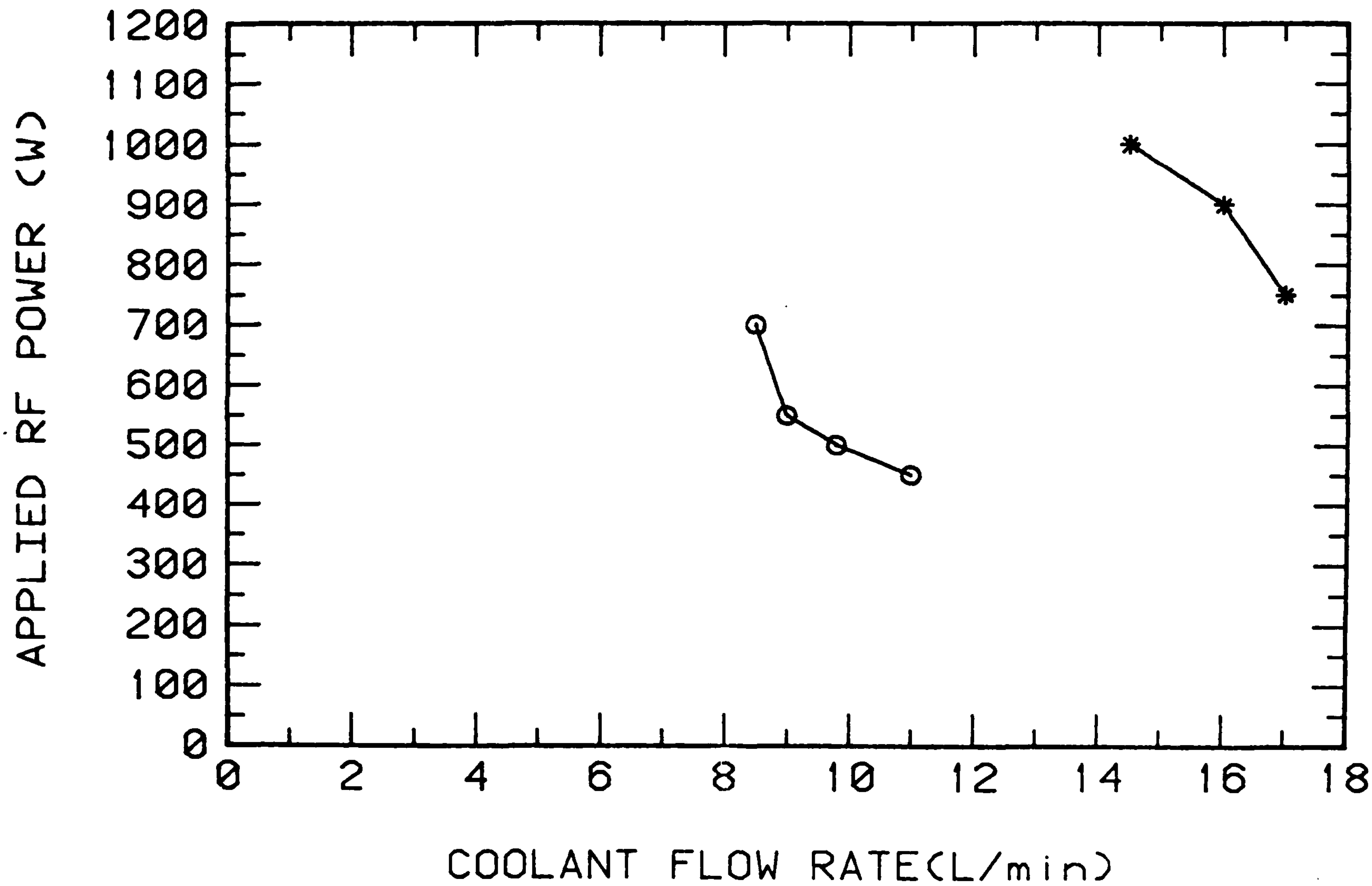
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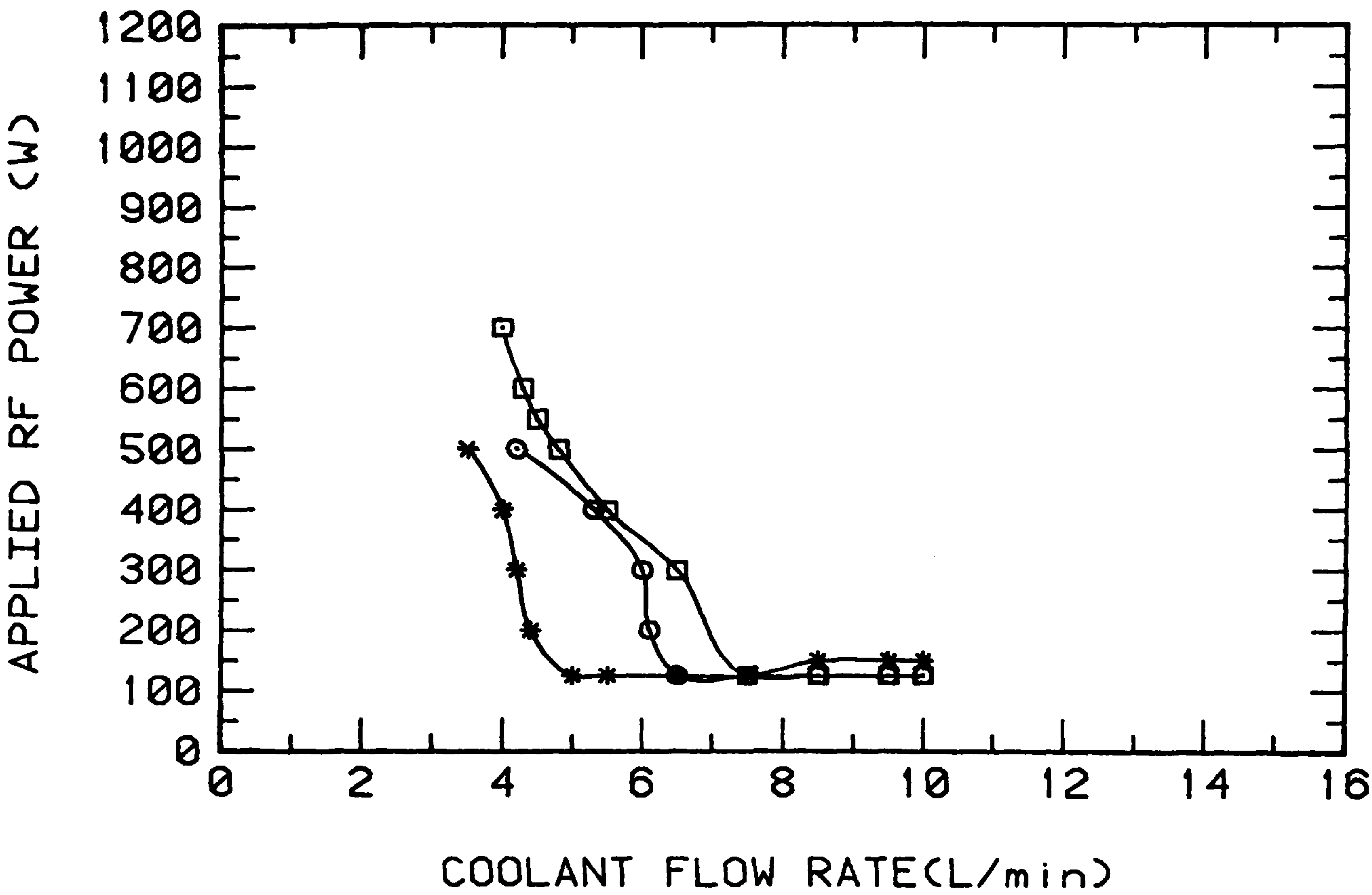

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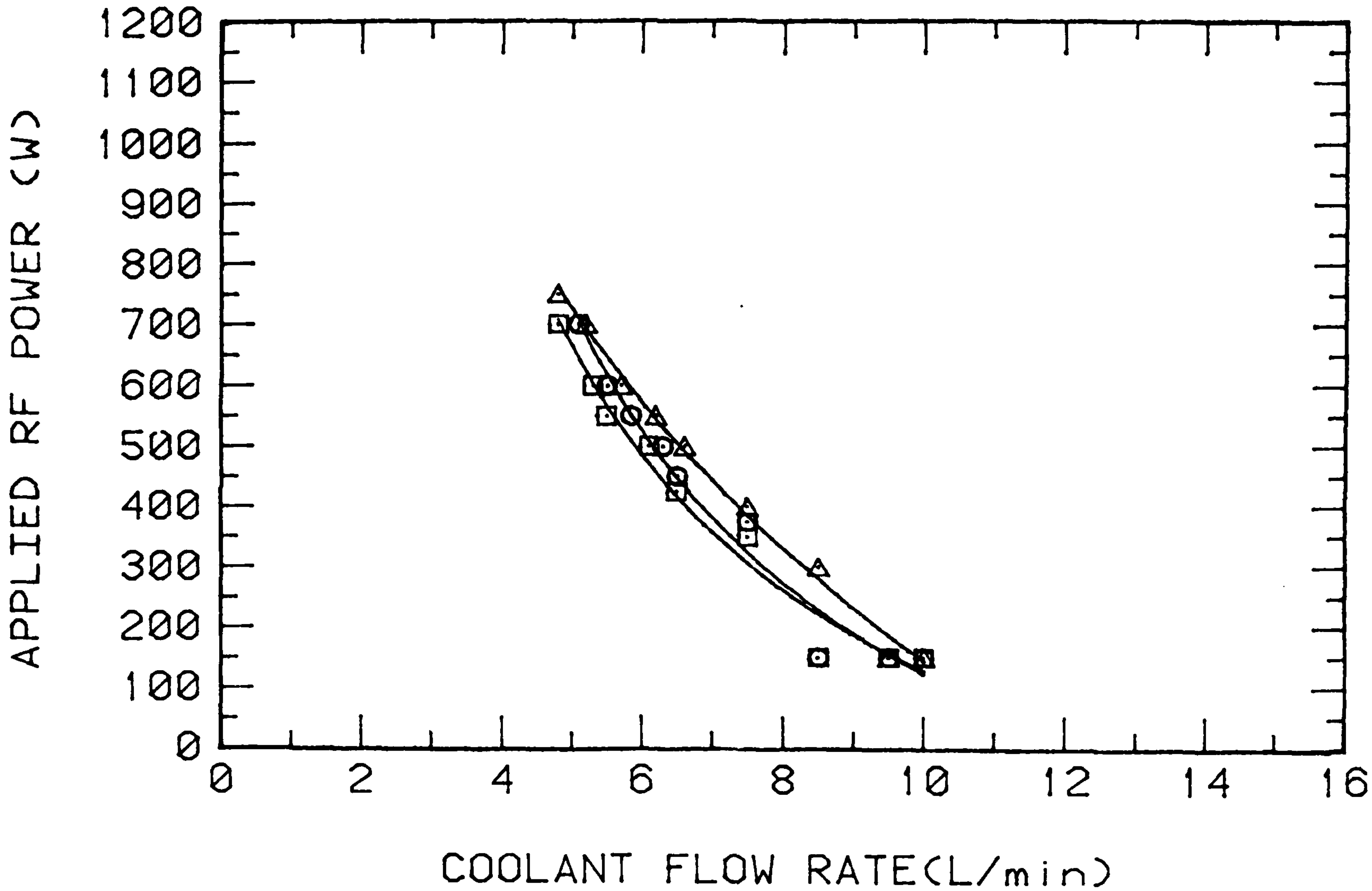
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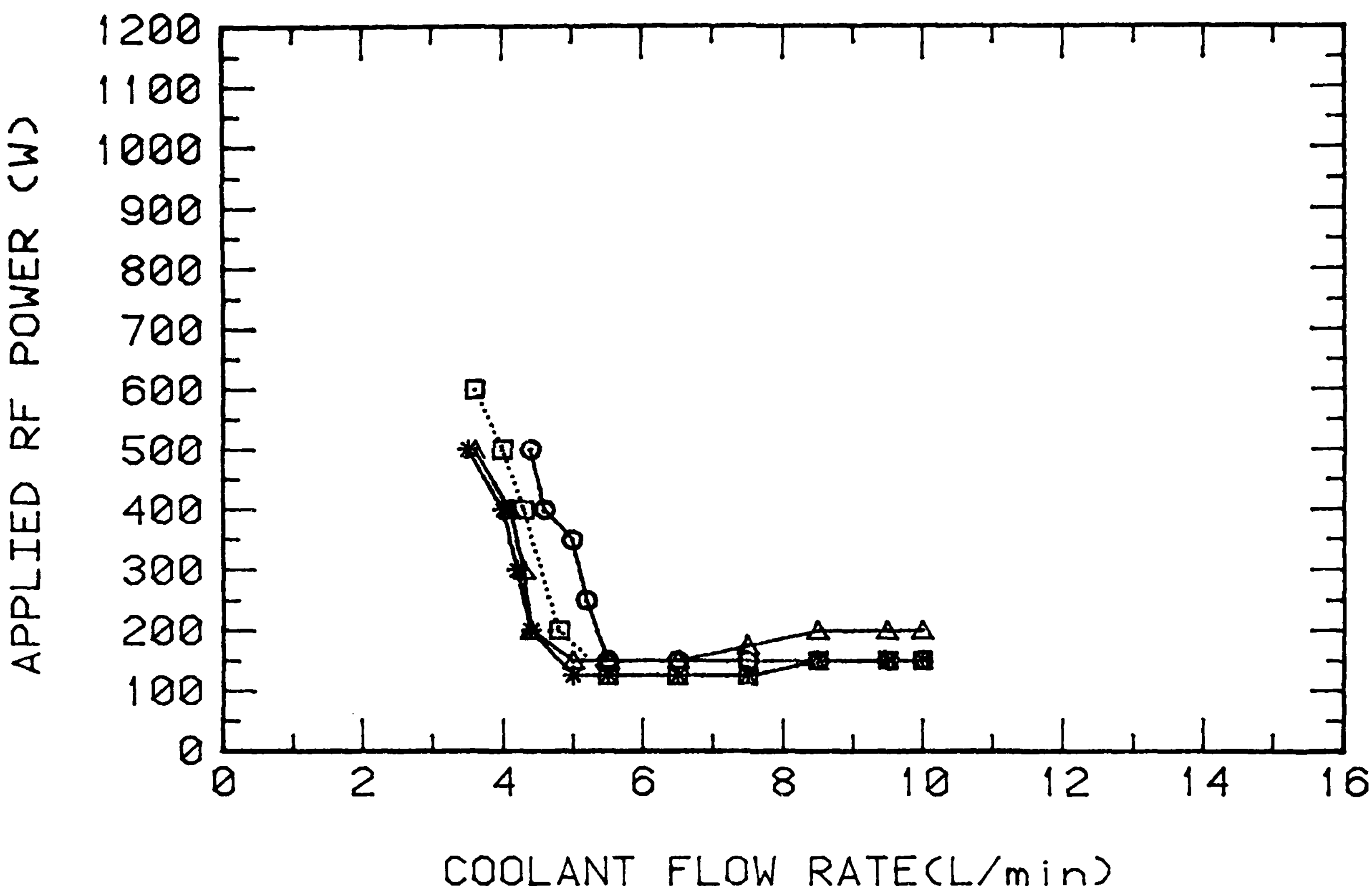
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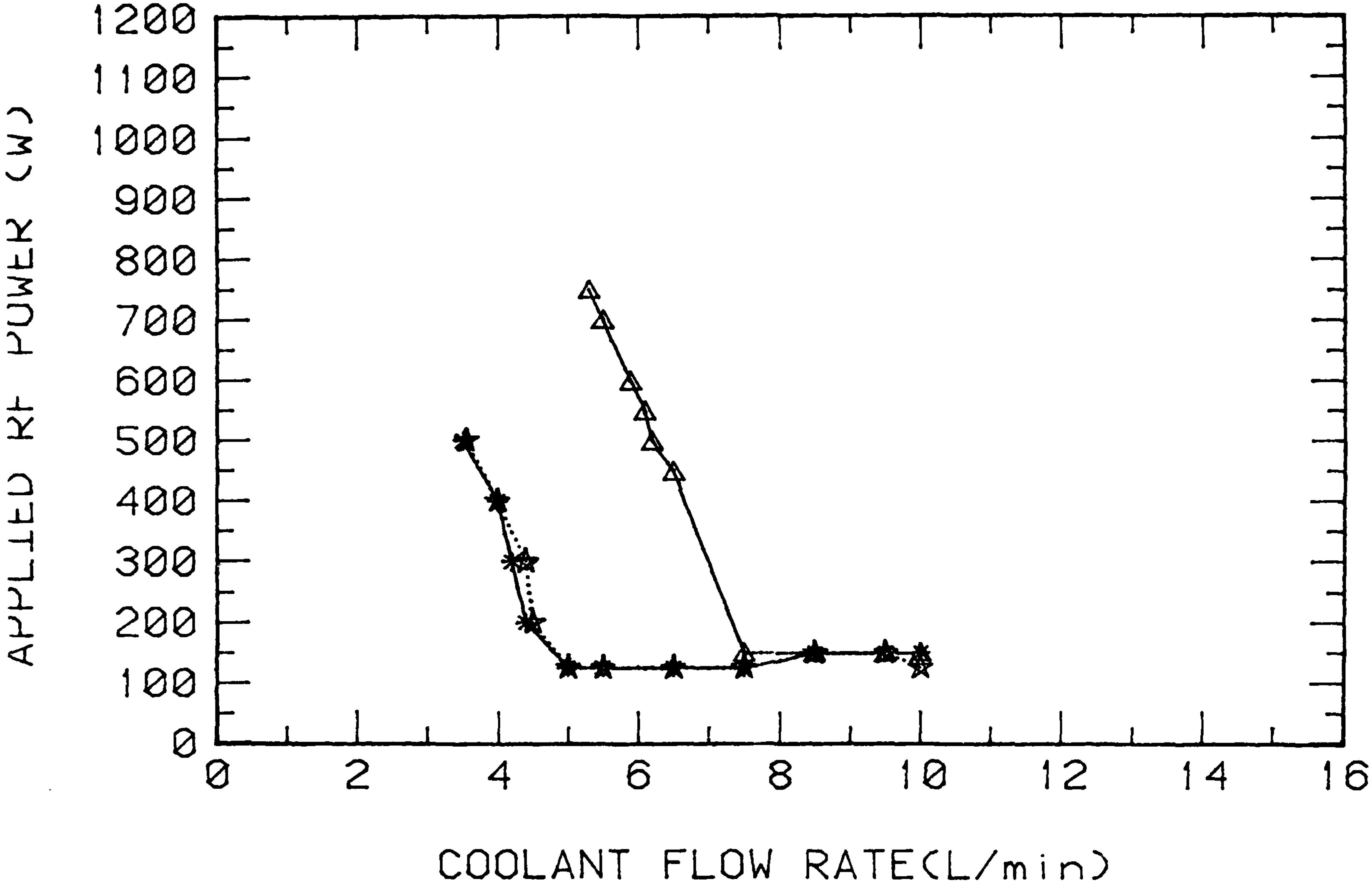

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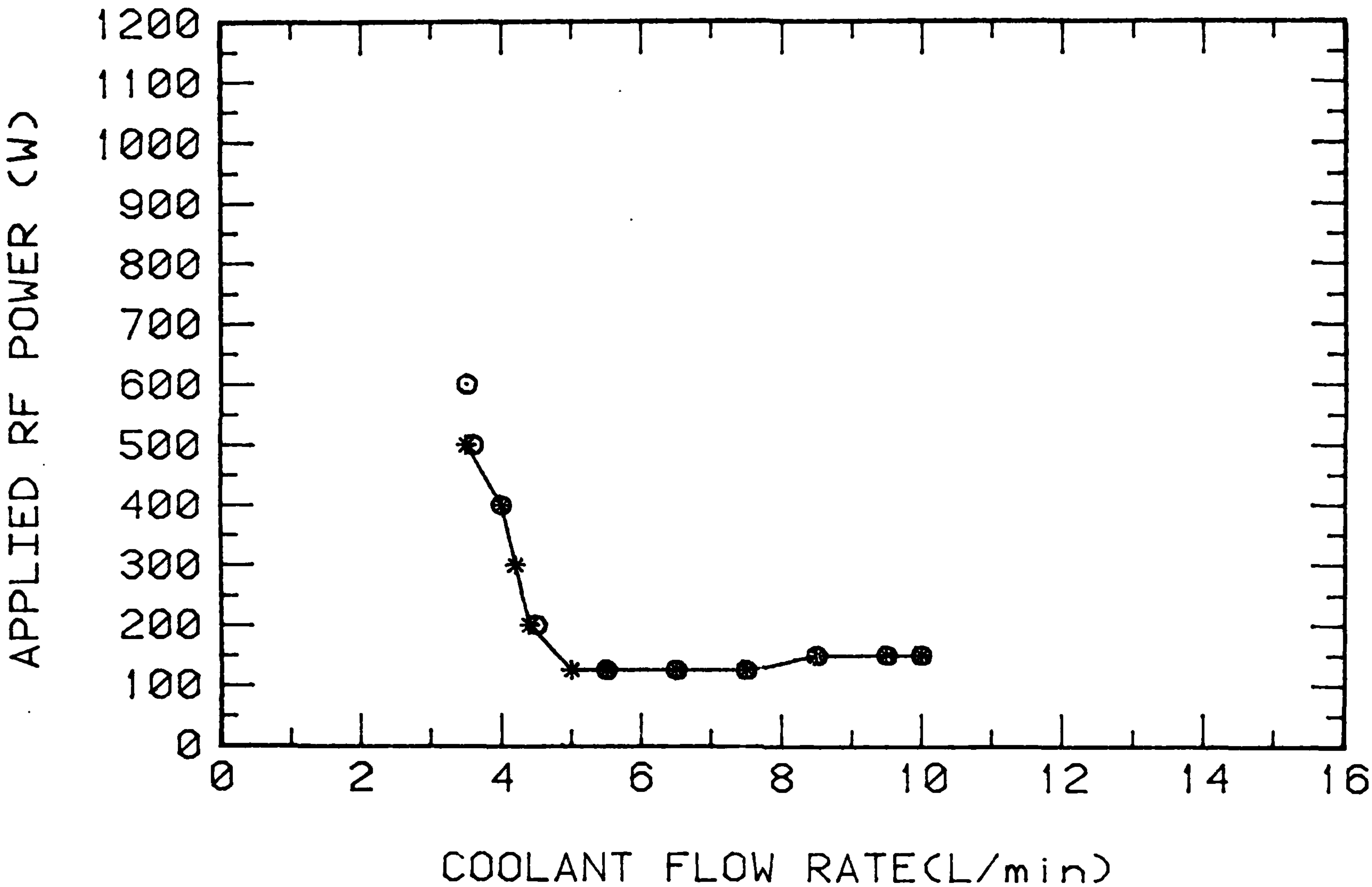
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